



Multiannual Programme of the Joint Research Centre 1980-1983

1980 Annual Status Report

High-temperature materials

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of the Joint Research Centre
1980-1983**

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High-temperature materials

Published by the
COMMISSION OF THE EUROPEAN COMMUNITIES
Directorate-General
Scientific and Technical Information
and Information Management
Bâtiment Jean Monnet
LUXEMBOURG

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© ECSC-EAEC, Brussels-Luxembourg, 1981
Printed in

ISBN
Catalogue number

HIGH TEMPERATURE MATERIALS

1980

Research Staff :

38 persons

Budget :

3,714 Mio ECU

Projects :

- 1. High Temperature Materials Information Centre**
- 2. Materials and Engineering Studies**
- 3. High Temperature Materials Data Bank**

Programme Manager :

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1. INTRODUCTION

The High Temperature Materials Programme is executed at the JRC, Petten Establishment and has for the 1980/83 programme period the objective to promote within the Community the development of high temperature materials required for future energy technologies.

The strategy of this programme is a combined approach to survey technology and industrial requirements in order to identify future needs, to establish and perform

research projects in selected key areas (coal conversion and related processes) and finally to build and maintain multilateral communications with manufacturers, users and also with the research and development sector involved in these materials.

The programme contributes to the public service and the "central nature" roles of the JRC by provision of scientific and technical information and expertise and also by acting as a focal point for co-ordination.

2. RESULTS

2.1 Information Centre

The objectives of this project are the provision of information service functions to the European HTM community and the encouragement of co-operative actions.

In order to meet these objectives, the Information Centre has undertaken three separate activities, i.e.

- Information Exchange and Transfer, organising conferences, symposia, colloquia, seminars and courses,
- Information Collection, executing inquiries, surveys and studies,
- Information Collection, establishing an Inventory on on-going research.

The results obtained are presented in Fig. 1.

2.2 Materials and Engineering Studies

In recent years there has been a considerable revival of interest in coal utilisation technology as the result of the increasing awareness throughout the world that supplies of oil and natural gas are rapidly diminishing, whereas coal supplies are plentiful.

This has prompted considerable effort to develop more advanced coal processing techniques. In most cases the main objective is to convert coal into forms of energy which are more convenient and clean to use; e.g. electricity, gaseous and liquid fuels and chemical feedstocks. At the same time the process must operate in an environmentally acceptable manner and should attain the highest possible conversion efficiency. The result of this is that plant components and construction materials are exposed to very aggressive environments at very high temperatures.

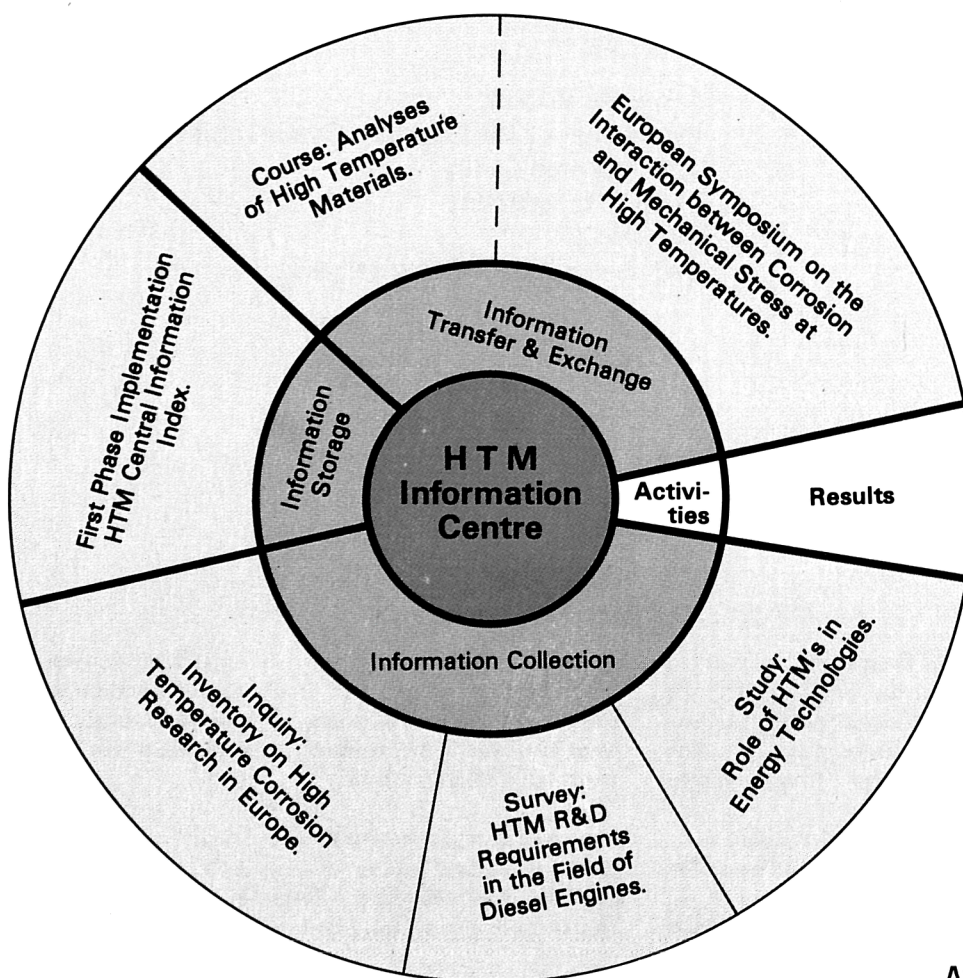


FIG. 1

**INFORMATION CENTRE
ACTIVITIES AND RESULTS.**

Similar types of corrosive atmosphere are found in a number of high temperature petrochemical processes where the severity of the environment is increasing as a result of the drive to higher operating temperatures for improved efficiency, and also to lower grade fuels for economy. The High Temperature gas cooled Reactor (HTR) developments provide further examples of critical application areas. A common feature of the numerous plant components in these various industries is the requirement for them to operate at temperatures in the range 700 - 1100 °C for long periods under stress, in aggressive environments which mostly have a restricted oxidation potential.

Therefore the aim of this project is to attain a deeper knowledge of the influence of different gaseous environments on the high temperature behaviour of selected superalloys by seeking to describe the parameters affecting time dependent mechanical properties and the kinetics of the corrosion which is occurring simultaneously during exposure in order to assist life-prediction studies of these components.

The type of gaseous environments being used during 1980 typify those found in various petroleum refinery, petrochemical, biomass conversion and HTR processes. They also assist in the interpretation of effects found in the more complex coal conversion atmospheres. In various applications the restricted level of oxygen can affect the development of a protective scale on the alloy. It is found, therefore, that there are life limiting problems, due to carburisation of the material or due to interaction between different corroding species, such as

oxidation and carburisation or sulphidation.

In the experimental work, therefore, high priority is given to understanding the relation between the micro-structural changes in the material and the behaviour observed during tests.

The alloys under test are, for the most part, the heat resistant steels which are typical or candidate structural materials for fossil fuel conversion processes. They fall into two groups with representatives in each of wrought and centrifugally cast products :

25%Cr - 20/25%Ni
e.g. Type 314, HK 40, IN519

22/25%Cr - 33/35%Ni
e.g. Alloy 800H, HP40Nb, HP30W

In addition certain alloys which are used predominantly in gas turbines are being examined either in the above environments or in air :

Nickel base
e.g. Hastelloy X, IN738 LC, Waspaloy

Cobalt base
e.g. HA 188

The investigations which, so far, have involved only air testing (Dynamic Loading and Coating) are being carried out in cooperation with COST 50 (European Co-operation in the field of Science and Technology - Action 50, Gas Turbine Materials).

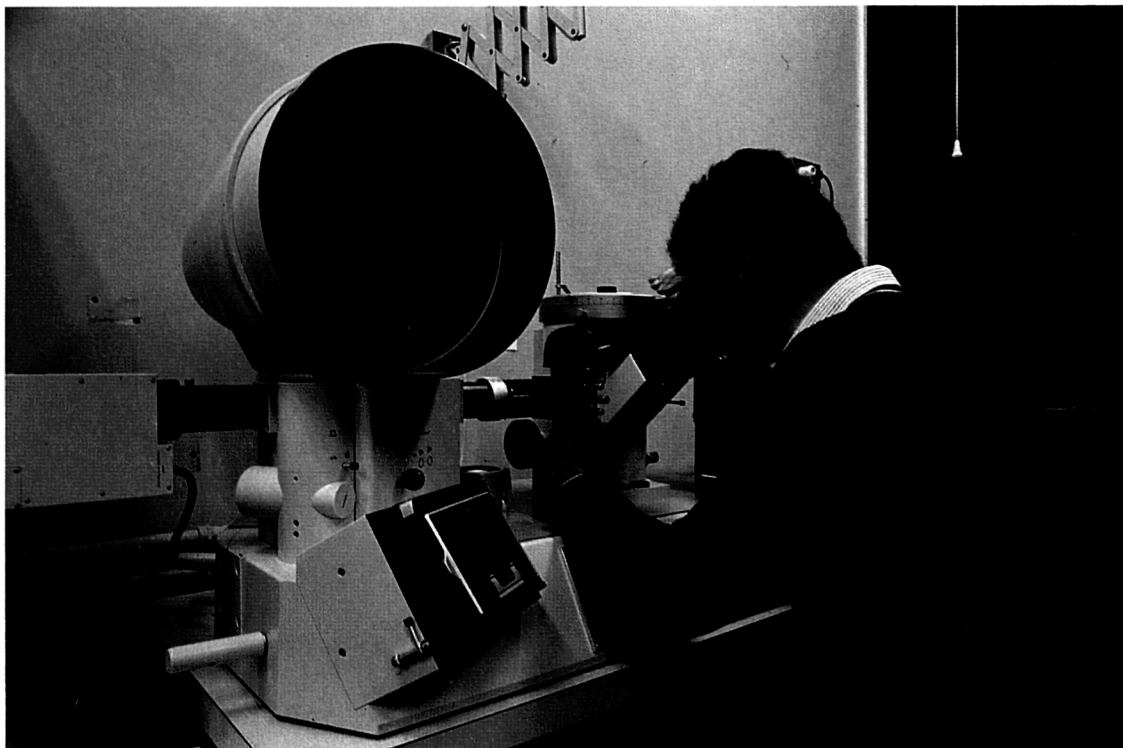


FIG. 2 OPTICAL MICROSCOPE.

The various detail activities which make up the work of the Project will be described below, having been arranged into an order which shows their interlocking nature.

Corrosion without Load

Environmental parameters such as temperature and the chemical activity of carbon (C), oxygen (O) and sulphur (S) are varied systematically and different alloy compositions are used so that a more scientific basis can be formulated for the selection and the continuing development of new materials to satisfy the demands of the service applications.

Intermittent weighing of the specimens during the periodic interruption of tests conducted in our autoclaves at temperatures between 800 and 1000 °C ensures that the kinetics of the corrosive reactions are monitored. The mechanisms by which the corrosive degradation proceeds are elucidated by detailed examination of representative specimens using surface

and cross-sectional structural analysis techniques, e.g. X-ray diffraction, optical microscopy, electron-microscopy and electron-spectroscopy.

A number of heat resistant austenitic steels together with a few nickel- or cobalt-base alloys are under investigation.

The comparative kinetic behaviour resulting from exposure to a highly carburising gas (hydrogen-methane) at 1000 °C shows the relative importance of various environmental and materials variables upon the carburisation behaviour. It was observed, for instance, that the tungsten containing iron-, nickel- and cobalt-base alloys exhibit a generally superior resistance to carburisation compared to the other materials.

The nickel : (chromium + iron) ratio has also been shown to be important in reducing carbon pick-up at high temperatures; increasing the ratio from 0,25 to 1,0 results in a 3 fold improvement in the time to absorb a given weight of carbon. In the same high carbon activity gas mixture, tests at 825 °C showed that increasing the Si level from 0,25 % to 1,5 % resulted in an order of magnitude improvement in corrosion resistance.

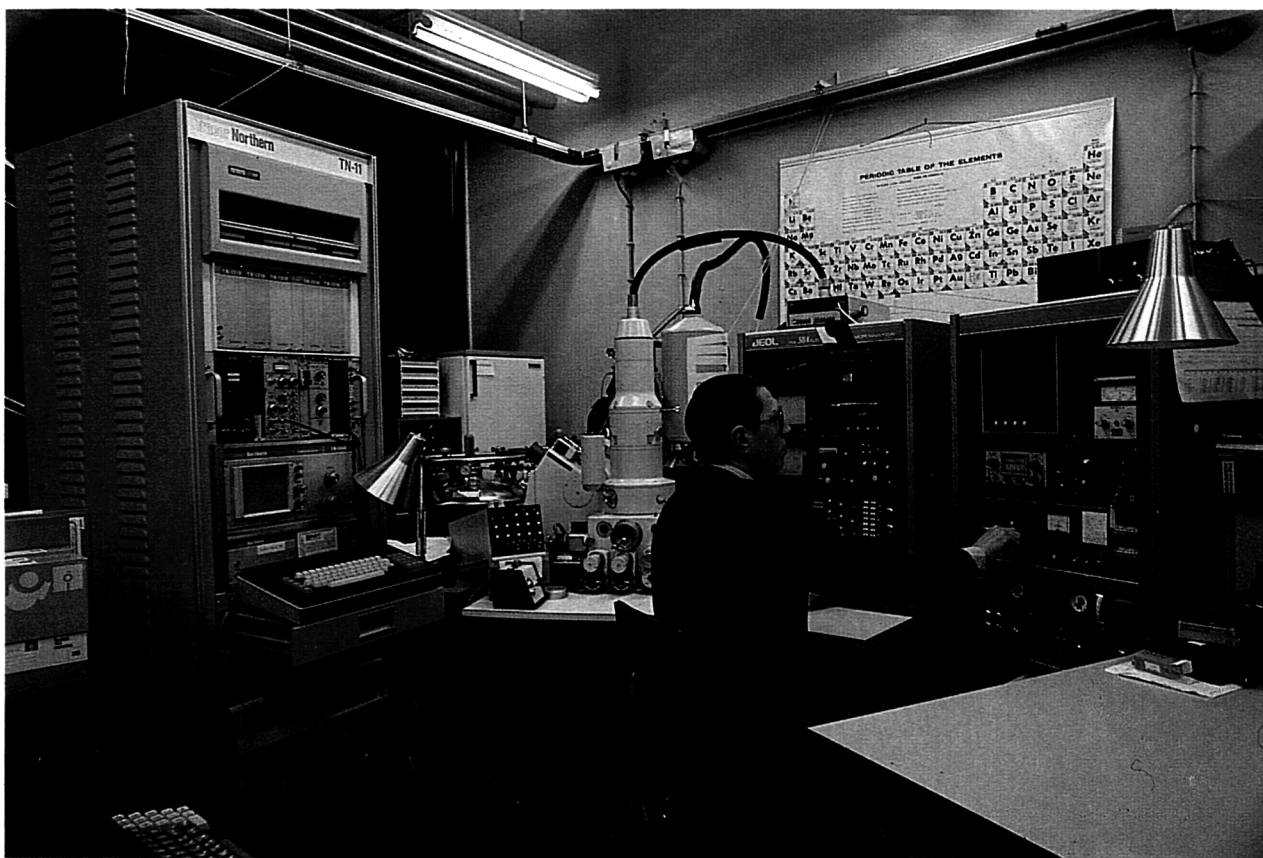


FIG. 3 EQUIPMENT FOR SCANNING ELECTRON MICROSCOPY AND ELECTRON PROBE MICROANALYSIS.

Corrosion work in more complex carburising/oxidising environments has continued with structural examination of specimens exposed to a pilot steam reforming plant, operating at $\sim 800^{\circ}\text{C}$ and 30 bar pressure and with laboratory exposures of similar specimens in a simulated steam reforming atmosphere. The latter samples have provided evidence of selective "carbon-dumping" from the environment. This appears to be catalysed by a higher nickel content in the alloy, surface finish also being contributory. As an example, Alloy 800H develops a surface layer of carbon when tested with an electro-polished, work-free surface, but not when the surface is mechanically worked (buffed or machined).

Test results are now available for the same range of materials exposed to sulphur-bearing atmospheres with a low chemical activity of sulphur, at 825°C . These conditions could be taken to represent "above bed" conditions in a conversion process using a low sulphur feedstock. A model alloy containing 50%Cr, 50%Ni, but no minor elements, suffered severe attack. However, the degradation on the various commercial alloys and the differences between them was much smaller. It was particularly observed that the nickel base alloy (Hastelloy X) was comparable in behaviour to the best iron based austenitic steel. Although test durations were short and significant degradation occurred, the rates

observed may be tolerable, so allowing these nickel containing alloys to be used where the sulphur activity is low enough to avoid the formation of nickel sulphide.

Corrosion under Load

The intensity of a corrosive attack on metallic materials by aggressive environments can be strongly stress dependent.

Conclusions derived from corrosion tests without load therefore need to be confirmed or modified for conditions where the materials are under load. This activity aims to obtain a qualitative understanding of corrosion mechanisms and some quantification of corrosion kinetics under the action of static and dynamic stresses.

Preliminary results from tests on two heat resistant steels (HK40 and Alloy 800H) in a hydrogen/methane gas mixture with high carbon activity at 1000°C showed that in the stress range corresponding to long life application (e.g. 5 - 10 years) the effect of creep deformation on carburisation kinetics is small.

A different conclusion might be expected, however, in a situation where a stable oxide could form on the metal surface.

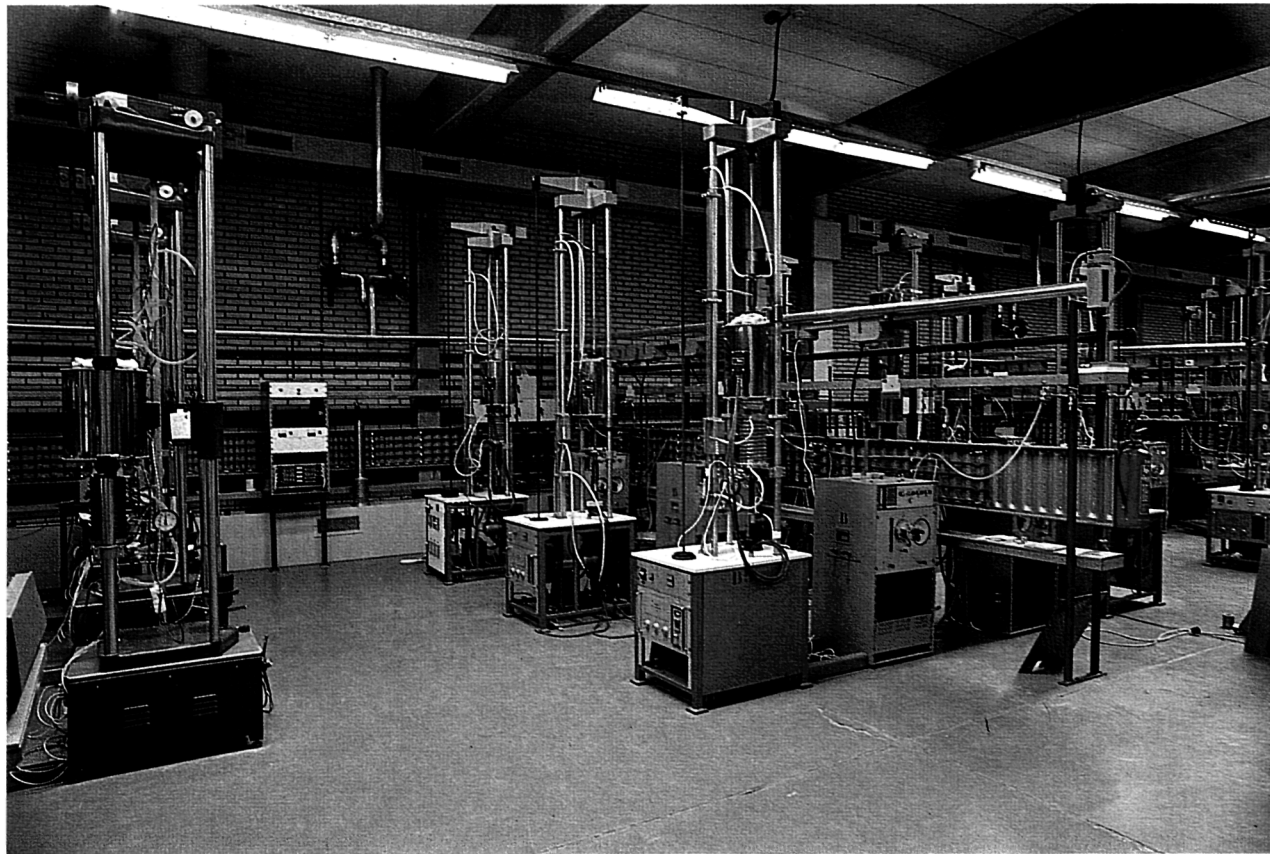


FIG. 4 CREEP TESTING UNITS IN THE ENVIRONMENTAL TESTING LABORATORY (ETL).

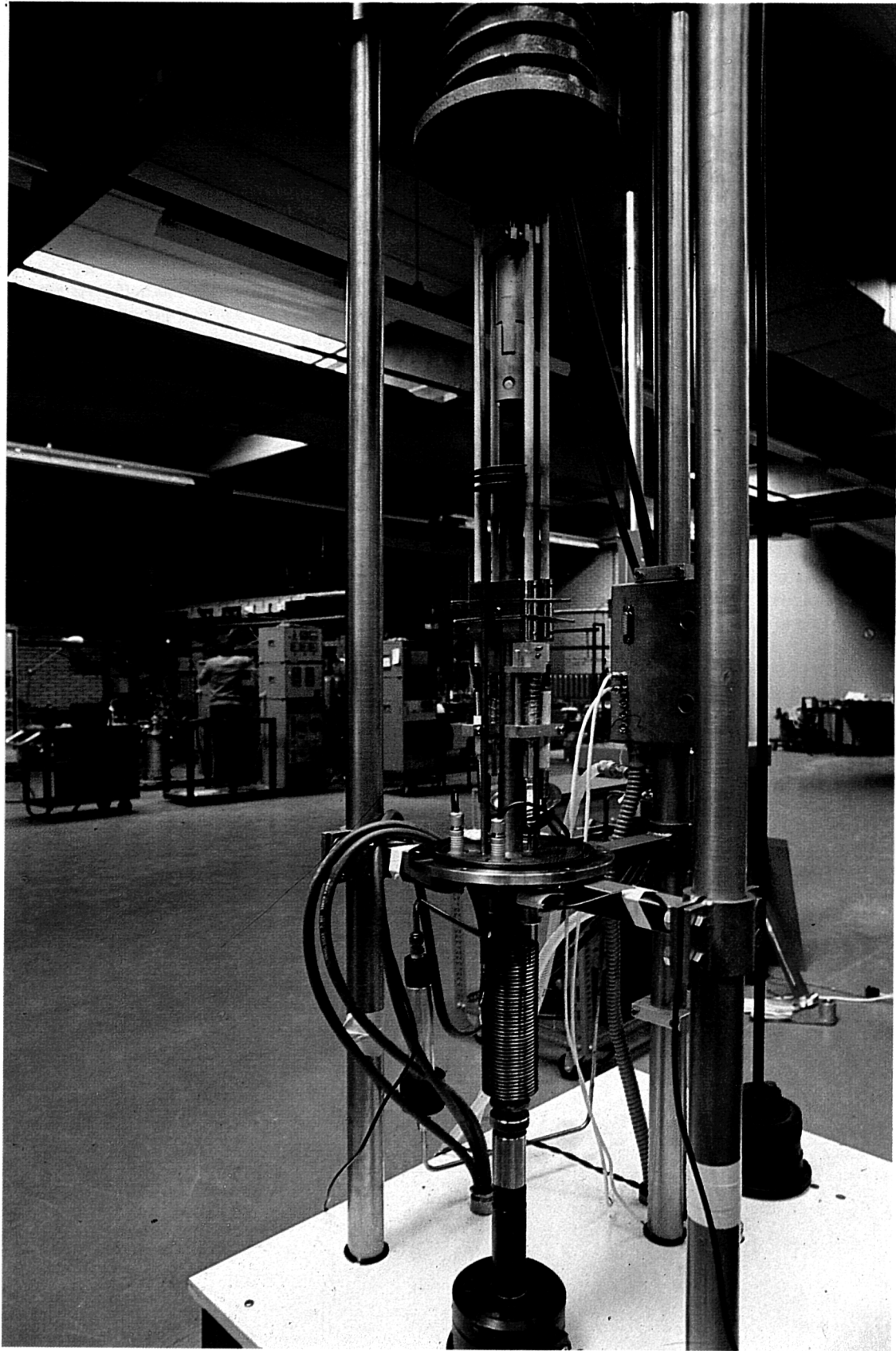


FIG. 5 DETAILS OF CREEP MACHINE IN ETL.

Mechanical Properties under Static Loads in Corrosive Environments

Special purpose creep test machines with environmental chambers, which have been developed in the laboratory, have been used to obtain high sensitivity creep curves up to fracture on materials which are concurrently exposed to a gas mixture of hydrogen/methane with high carbon activity at 1000 °C. This has been shown by consideration of the industrial applications and the corrosion test results to give the most suitable and severe environmental conditions for degradation by changes in the creep deformation processes. Tests have also been performed to obtain similar data on the same material samples in air at temperatures between 900 °C and 1100 °C. This forms the basis for assessment of environmental effects on creep and rupture properties.

Two alloys have been selected for detailed study following consideration of the industrial situation and the corrosion behaviour. They represent commonly used tubes made by centrifugal casting (HK40 in low and high carbon versions) and forging (Alloy 800H).

At the relatively high stresses which are sufficient to cause failure of HK40 in < 1500h, the creep rupture strength is reduced by testing in the corrosive gas; the maximum effect being a factor of 2. A similar, but less pronounced situation is found when the test piece is fully carburised before testing. At stresses low enough to cause failure after > 1500h the test piece used becomes through-carburised during testing and the creep rupture strength is raised progressively above the air data. The trend to improved life is related to the

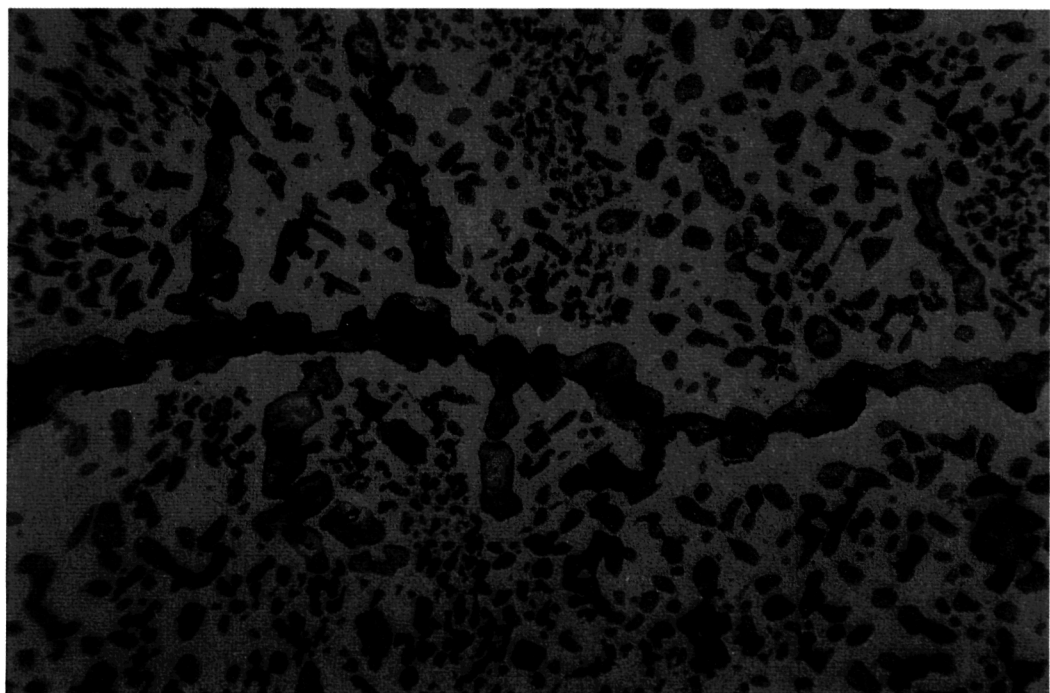
increase that takes place in rupture ductility which in turn is the result of microstructural changes that take place during carburisation. At long durations the increase in rupture ductility is threefold (3 % to 10 %).

In contrast, the creep rupture strength of the other major material, Alloy 800H, is increased at all applied stress levels as a result of progressive carburisation. This is particularly noticeable at lower stress levels (longer life tests, greater carburisation). The rupture ductility remains high at all times. Fully pre-carburised specimens, tested in carburising gas, reveal a further increase in ductility compared with the normal progressive carburising tests; the maximum test duration in the test series being > 4000 h.

Microstructural analysis reveals that at high stress levels (~ 100h life) both HK40 and Alloy 800H develop wedge shaped cracks at grain boundary triple-point intersections, while at lower stresses round intergranular cavities are formed. Cavities and cracks are invariably initiated at the interface between carbide particles and the matrix austenite.

An important observation has been that during testing of HK40 with progressive carburisation, large cavities and cracks can form in the uncarburised core at an early stage as a result of strain accumulation due to summation of the applied stress and that resulting from the dilation due to carbon ingress.

These cracks remain stable when the carbon front reaches and engulfs them (see Fig. 6). Failure only occurs after subsequent advancement of the creep process.



**FIG. 6 CARBURISED INTERGRANULAR VOIDS IN HK40 CREEP
TESTED AT 1000 °C IN HYDROGEN/METHANE GAS. (450x)**

This pattern of behaviour is consistent with the observations made on failed thermal cracker tubes. It can be concluded that no effects should be seen in long life components as a result of their exposure to a carburising environment at about 1000 °C which would be deleterious to their life at this temperature. The well known embrittlement occurring at room temperature must not be neglected however.

Scatter in Creep Rupture Behaviour

Examination of creep and rupture data frequently shows that there is considerable scatter in results originating from variability in test parameters between tests and in metallurgical variation between test pieces. Thus to explore metallurgical effects meaningfully, it is necessary to have adequate control over test parameters.

In order to quantify the contributions from these two sets of variables on the observed scatter in creep properties, the behaviour of one cast of Waspaloy, a typical nickel-base superalloy, was explored. It was concluded in the first part of the work that test parameter variability on the scale which is expected to apply under conditions of normal laboratory practice may substantially contribute to scatter of stress-rupture properties. In terms of rupture times, for example, a scatter of 50 % to 100 % can be caused by a combined variability of stress and temperature. Precise control of test technique variables, as is practiced in this laboratory for all creep testing reduces the scatter to an insignificant level, however.

Using this close control of test parameters, the variability in high temperature creep behaviour of the cast due to metallurgical factors was explored. It was concluded that scatter in the primary and secondary stages of creep was small. Only in the tertiary stage was significant scatter observed, with a clear correlation between rupture life, t_r , and tertiary strain, ϵ_t .

Creep damage develops during the tertiary creep stage with the formation of intergranular wedge-type cracks and surface cracks. An increase in the rate of formation and propagation of these cracks results in a decrease of ϵ_t and consequently a decrease of t_r .

The scatter in ϵ_t and t_r correlates well with the difference in grain boundary chemistry between the respective specimens. Auger electron spectroscopy reveals that in short life samples, the grain boundary concentrations of sulphur and oxygen are significantly higher than in long life samples. This variability can be expected to result from ingot segregation patterns and also from the non-uniformity in thermo-mechanical treatment which is associated with normal commercial practice.

Creep of Tubular Test Specimens in Corrosive Environments

The planned goal of this work is to perform multiaxial creep experiments on tube shaped specimens in corrosive environments at high temperatures. Such complex high pressure tests require sophisticated experimental equipment and infrastructural facilities, but they help to bridge the gap between the knowledge of materials behaviour gained from conventional specimen tests and that required for engineering applications.

Alloy 800H tubes with 4-5mm wall and 33mm outside diameter have been tested in uniaxial creep, to compare their behaviour with that of the laboratory testpieces described above. Although the scatter of results is much greater for the large tubular specimens there is good agreement in terms of time to failure under constant loads at temperatures of 900 °C and 1000 °C in air.

Internal pressure tests on Alloy 800H tubes with the same cross section have been carried out using argon as the pressurising medium. All tubes failed under hoop stress with a lower ductility than the longitudinally stressed specimens, but their life to rupture was similar to the uniaxial results as might be expected for a wrought homogeneous material.

Special effort was also put into all aspects of the design of an installation to house a few tube testing units which will be able to apply longitudinal loads to tubes under internal pressure of a corrosive gas at high temperature. This multiaxial corrosive creep testing facility will provide, for small diameter tubes, a simulation of components for various high temperature industrial processes.

Study of a High Temperature Test Facility for Tubular Components

In order to explore Community needs for data on the performance of plant items, the specification of a tubular component testing programme, which would be carried out in a large advanced test facility was realised during the year. The proposed programme would evaluate the performance of critical sub-components such as pipes, bends, intersections and welded tubes under experimental conditions simulative of service as seen in crackers, reformers, high temperature reactors, and coal conversion plant. A large facility required to carry out the programme was also proposed. It must be pointed out that such facilities are very expensive to realise both from the construction and operational points of view, essentially because of the safety requirements. The advice of the programme advisory body concerning this investigation was to draft a proposal for a small project to be included among the proposals which will be submitted for execution in the next programme period.

Mechanical Properties under Dynamic Loads

A reasonable level of knowledge already exists concerning the behaviour of many materials under static loads in oxidising environments.

With regards to the varying load situation which results in fatigue, the level of understanding is much lower, although it is more frequently encountered in practice. The introduction of aggressive environments complicates the situation even more and the resulting situation is characterised by a multitude of simultaneously operating processes in the material which may or may not mutually interact.

The initial work for the exploration of the creep/fatigue interaction to be expected in High Temperature Low Cycle Fatigue (HTLCF), was orientated towards the behaviour of gas turbine disc forgings. An air environment is appropriate for these tests. Because of its application area this investigation has been integrated into the COST 50 activities.

Test pieces of the nickel-base alloy Waspaloy were subjected to low cycle fatigue at 500 °C and 650 °C to assess the effects of strain rate, strain amplitude and temperature on the cyclic life of the alloy at temperatures at or above the maximum found in service.

Increase of temperature from 500 °C to 650 °C results in a reduction in cyclic life, the magnitude of which depends upon strain rate and amplitude, the greatest effect being seen at slow strain rate and small amplitude. At constant temperature and constant strain amplitude, the cyclic life is again decreased with decreasing strain rate to a minimum value at $\sim 10^{-4} \text{ s}^{-1}$ (0,1 Hz for a "service" strain amplitude) at which a saturation level is reached. Microstructural examination of failed specimens reveals a deformation mechanism of planar slip. Crack propagation is purely transgranular at high strain rate and low temperature. Only at 650 °C and the slowest rate ($\dot{\epsilon} = 5 \times 10^{-5}$) does intergranular cracking appear.

From these results, it appears that in addition to cycle dependent fatigue there are at least three active time dependent processes:

- i) hardening of the alloy due to dynamic strain ageing, the extent of which varies with cycle mode, strain amplitude and rate;
- ii) oxidation ahead of the crack tip which reduces alloy strength by inward diffusion of oxygen and outward diffusion of reactive metals, and
- iii) intergranular creep deformation of the alloy at higher temperatures and slow strain rates resulting in grain boundary weakening by wedge crack formation.

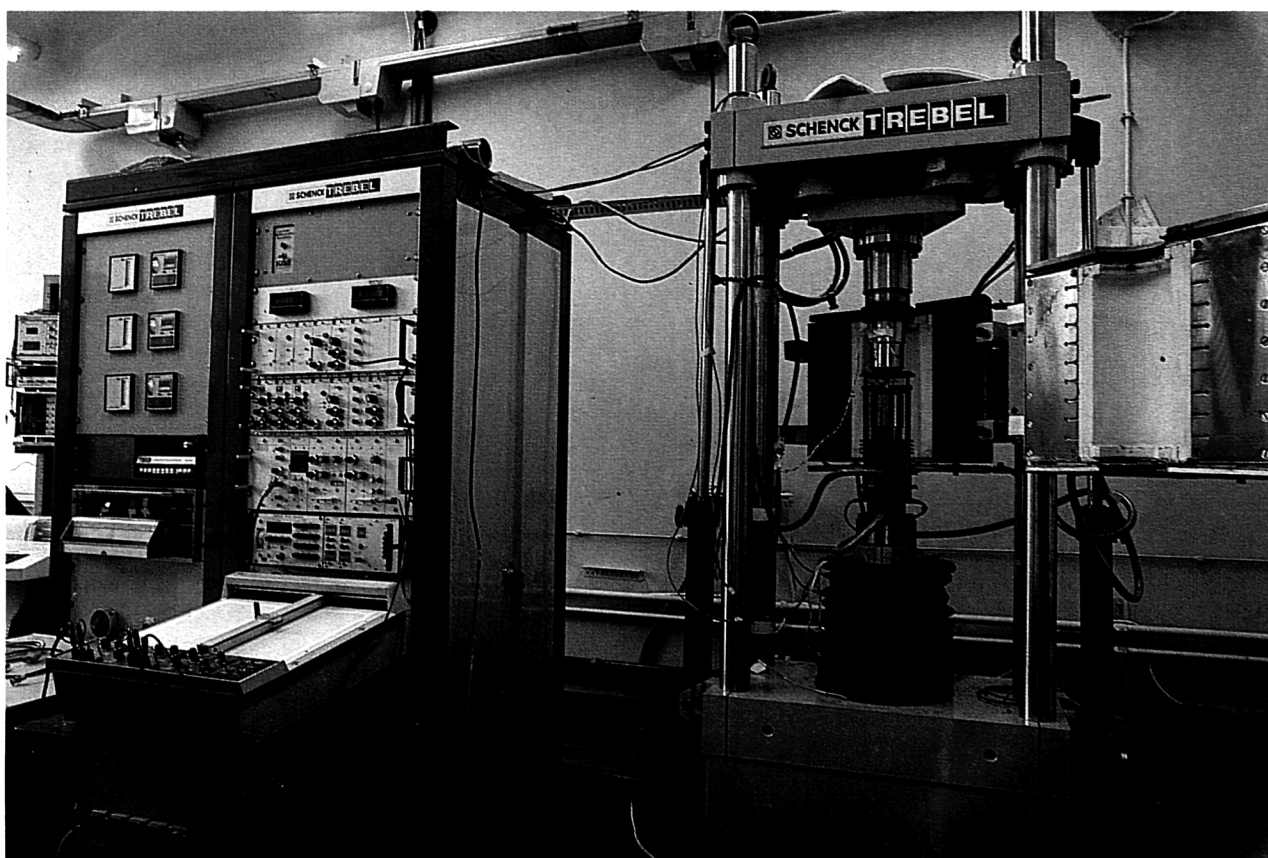


FIG. 7 EQUIPMENT FOR HIGH TEMPERATURE LOW CYCLE FATIGUE TESTING.

Work is now proceeding on creep-fatigue interaction in another nickel-base alloy, Powder Metallurgical Astroloy. This has confirmed the very significant and complex influence of strain rate and amplitude, on the temperature dependence of cyclic life. Thus at high strain rates and small amplitude the reduction in cyclic life on going from 400 °C to 730 °C is small, while at slower strain rates (e.g. $\dot{\epsilon} = 5 \times 10^{-5} \text{s}^{-1}$) for the same amplitude, cyclic life is reduced by a factor of 40. These findings emphasize the importance of testing at the very slow strain rates and amplitudes encountered in operational service. This part of the investigation has not yet advanced sufficiently to discriminate between the influences of creep and other processes on the observed results.

Surface Protection - Scales and Coatings

The use of all metallic materials for applications at high temperatures in aggressive environments requires the formation and preservation of a protective surface oxide layer or scale to act as a barrier.

In several engineering applications the strength requirements involve the use of materials which do not develop a protective oxide scale. Hence coatings able to withstand the conditions seen in service have to be used. The protective action of these coatings against corrosion is related to the development of their natural scales.

Common to both modes of surface protection is the need to preserve the oxide scale under the operating conditions. In addition to chemical reactions there are also mechanical constraints through oxide growth stresses, thermal cycling and/or superimposed mechanical loading which can cause fracture or spalling of the scale, so making it ineffective as a corrosion barrier. In the case of applied coatings, reactions may also occur between coating and substrate alloy which can detrimentally affect the protective ability of the coating and also the mechanical properties of the composite material.

The scope of this activity is to examine the behaviour of a number of oxide scales and protective coatings, on high-temperature alloys, to allow the selection of alloy/oxide systems which are capable of providing extended service in engineering applications.

Up to and including 1980, the work on the properties of oxide scales and the behaviour of coatings have been separated in experimental details; the former has considered scales forming on heat resistant austenitic steels while the latter has dealt with coatings applied to gas turbine blading alloys. As such it has been integrated into the COST 50 activities.

The investigation of factors affecting the oxidation kinetics and spalling behaviour of scales has concentrated on a series of model alloys based upon IN519 with varying Silicon (Si) and Yttrium (Y)

content, which were oxidised in the range 850 °C to 1000 °C. The model containing the same Si level as found in commercial IN519 (0,7 % Si) exhibited the greatest oxidation resistance, while low Si or Si-free alloys all suffered breakaway oxidation during the first 40h of exposure, forming thick scales which spalled on cooling. Additions of Yttrium provided little benefit to alloys containing less than 0,7 % Si but at this level improved adhesion under thermal cycling conditions was found. Measurements using a resonant frequency technique suggest that under isothermal conditions, all of the IN519 alloys grow strong, adherent scales and that stress build-up in the scale occurs at a steady rate. This contrasts with the behaviour of Alloy 800H, where the frequency-mass curve shows a sharp break. Microstructural examination suggests that this break results from a change in the interface roughness and therefore the adhesion between the scale and substrate.

The work on the IN519 series of alloys suggests, however, that the expected "keying" of the scale to the substrate has not been obtained. When this conclusion is combined with the difficulty of adding Yttrium to a centrifugally cast alloy, it seems that no commercial advantage would be gained. Some other, more advanced production route would be required.

Assessment of the effect of coating/substrate interactions has been carried out on the cast nickel-base alloy IN738 LC and has been conducted following reports of degradation in strength properties as a result of the formation of intermetallic phases at the interface between the coating and the substrate blading alloy. Conventional aluminide coatings, applied by pack diffusion processes (e.g. LDC-2) and new overlay coatings deposited by techniques which are still under development (e.g. low pressure plasma spray and ion plating) have been explored. The overlay coatings were in a NiCoCrAlY alloy identified as S57.

The creep behaviour of coated IN738 LC testpieces was compared at 850 °C to that of bare testpieces and it was found that there was no significant influence on the creep properties of the substrate alloy, either by the coatings or by their application procedures.

Subsequent metallographic analysis revealed that the predominant failure mechanism in all test pieces involves the formation of large round intergranular cavities which link to form internal cracks. The coatings displayed greater ductility than the substrate alloy and appear to inhibit surface cracking. In testpieces coated with S57, the quality heat treatment promotes the formation of a thin zone of complex structure, just beneath the initial specimen surface as a result of a chemical interaction between coating and substrate. Subsequent ageing, both with and without stress, results in some broadening of this interdiffusion zone and in the appearance of additional phases within it. However, this feature does not appear to influence the deformation behaviour or the development of cracks in the substrate alloy.

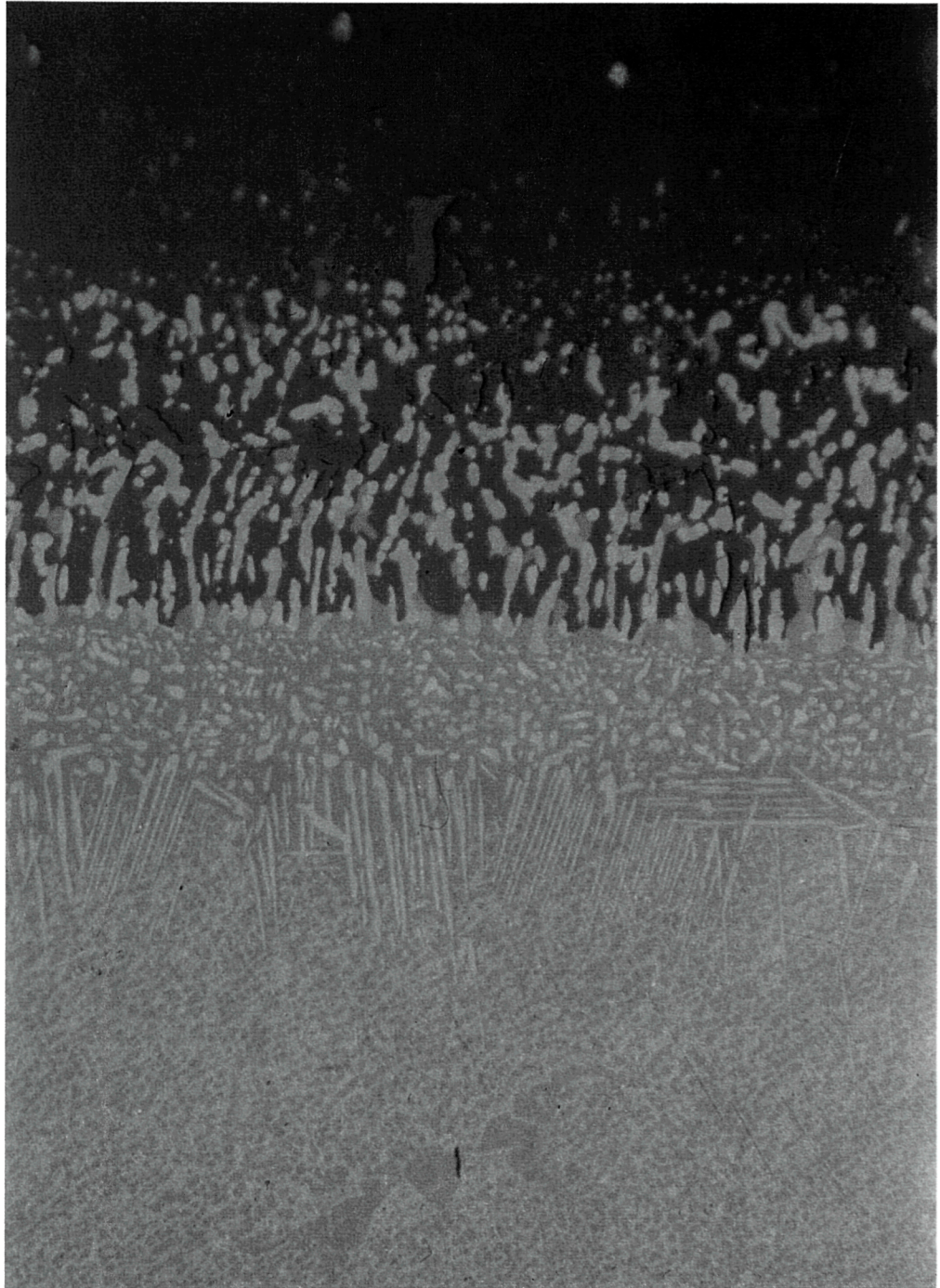


FIG. 8 COMPLEX COATING-SUBSTRATE INTERFACE STRUCTURE FORMED BY AGEING LDC 2 ALUMINIDE COATING AT 950°C.

Concurrently, ageing studies have been carried out on coated samples to explore the interdiffusion zone in more detail. This stress-free ageing study was expanded to include several other aluminide-type coatings. The development of platelike precipitates of intermetallic compounds occurred in every combination. These precipitates were identified as being mainly σ -phase although small quantities of another phase, ("R"-phase) were also detected. The development of these brittle phases could, potentially, have a detrimental effect on

thin section components, and this feature will be examined shortly in the planned thermal shock testing of tapered discs.

Reports from pilot coal conversion plants suggest that there are cases where the corrosive nature of the environment is so severe that coated components will be essential. Accordingly, this activity is being translated into a study of the behaviour of suitable coatings applied to some of the austenitic steels described in earlier sections.

2.3 Data Bank

In the third project the implementation of a pilot data bank on mechanical and corrosive properties of materials relevant to energy conversion technologies is undertaken. This data bank is planned to become a tool for research management in relation to data generation as well as an instrument for service to materials' users and producers. Such functions are now facilitated by the establishment of information networks for public use, like EURONET, which permits Community wide access to any type of data base associated with the system.

The programme has investigated the European interest for a data information system on high temperature materials and concluded that there is an interest for application-oriented, evaluated property data, which is at present not satisfied by any generally available information system existing in Europe.

The project started work relating to HTM data with a restricted scope of alloys and properties. Materials and properties of primary concern are those investigated in the HTM programme, in particular Alloy 800 and

tensile, creep and fatigue properties at 600 °C - 1000 °C in environments of C-O-H composition.

The data bank is developed in collaboration with the computing centre of the Ispra Establishment (see Fig. 9) where the available data base management software (ADABAS) is adapted to the data bank requirements defined by Petten.

The structure and contents were defined and thus the design basis for data collection and input procedures established. The first data input programme transforming data into the computer processing format was written and tested. The further development of the input programme is going on, using programming techniques closely allied to ADABAS. First experience on data collection has shown that the majority of data is to be collected from published literature sources and a minority by compiling experimental data from the Petten HTM programme or from co-operating partners. A trial run of a data collection was conducted, data analysed and a data searching and collection procedure designed. The final development of this procedure and the design of the data collection forms are in an advanced stage.

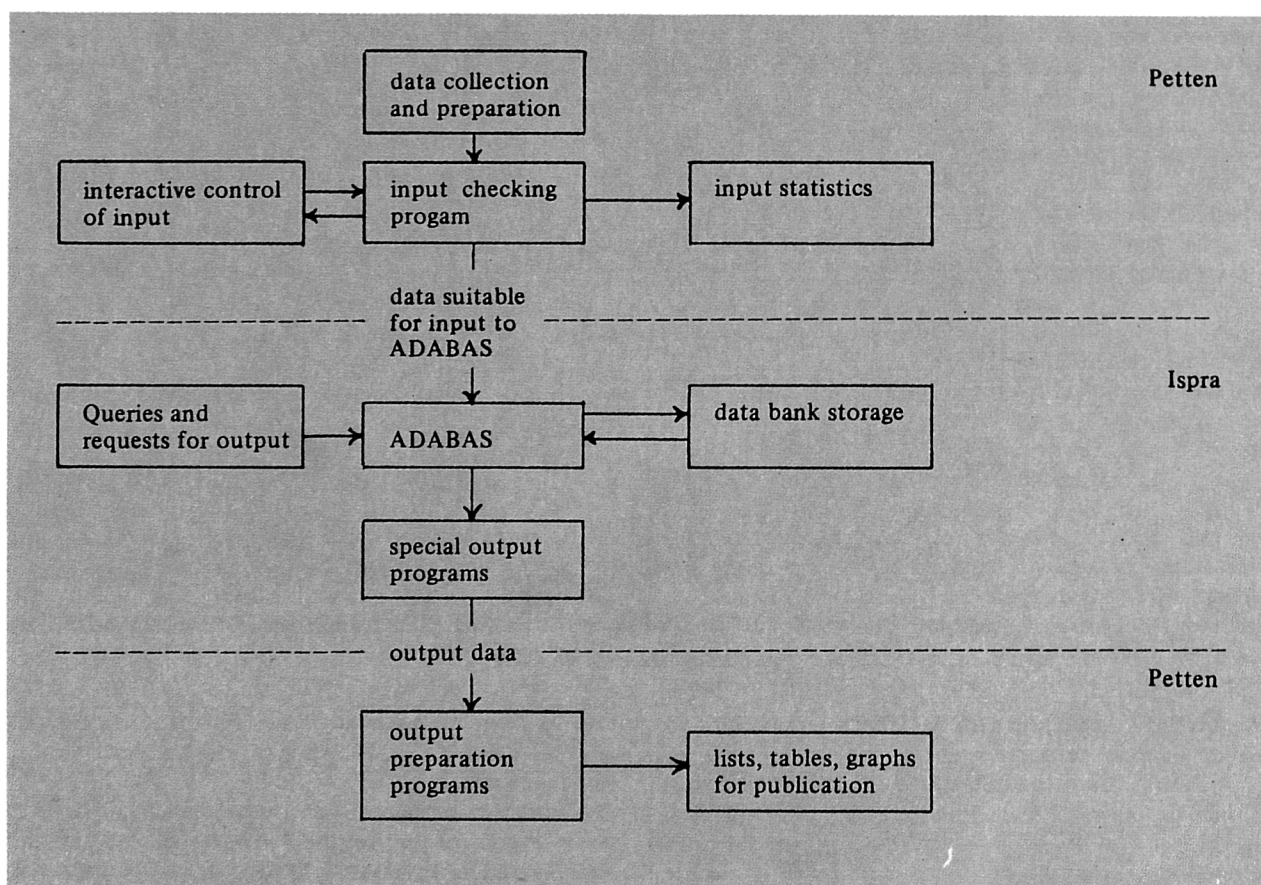


FIG. 9 HTM DATA BANK, SYSTEM PLAN.

3. CONCLUSIONS

The HTM programme contributes to developments in the new energy conversion field through the provision of improved knowledge on materials science and technology. Its activities were executed in close contact with universities, industries and governmental bodies. The concerted European action (COST 50) provided the frame within which the gas turbine research work was integrated.

- The Information Centre project was successful in supplying information on materials R & D and developing close contacts with national and industrial energy programmes making use of high temperature materials.
- The control of materials behaviour in corrosive environments is an essential pre-requisite for the realisation of advanced energy technologies. The programme has made significant efforts to study the

behaviour using a facility which is unique in its ability to test alloys at high temperatures in those toxic and explosive environments which simulate the corrosivity of environments found in fossil fuel conversion processes.

The results obtained increase the understanding of materials behaviour, assist the selection of high temperature materials for plant design and contribute to the development of new materials.

- The testing of tubular specimens is being conducted to effect a stronger link between the materials understanding and engineering aspects.
- The Data Bank is being successfully set up. When operational it will be an instrument for service functions to external users as well as providing a tool for research management in relation to data generation.

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